

GPO PRICE \$ _____


CFSTI PRICE(S) \$ _____

Hard copy (HC) \$ 1.00

Microfiche (MF) 150

7 653 July 65

NASA TT F-8478



METHOD OF FABRICATING SILICON CARBIDE SHAPES

by

Eduard Enk and Julius Nickl

Translation of "Verfahren zur Herstellung geformter Körper aus Siliciumcarbid"

German Patent 1,088,863, September 8, 1960


N66 27044

FACILITY FORM 908	(ACCESSION NUMBER)	(THRU)
	5	1
	(PAGES)	(CODE)
	15	(CATEGORY)
	(NASA CR OR TMX OR AD NUMBER)	

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

WASHINGTON

August 1963



FEDERAL REPUBLIC OF GERMANY

Cl. 80 b 8/11
Intern. Cl. C 04 b

GERMAN PATENT OFFICE

AUSLEGESCHRIFT 1 088 863
W 23078 IVc/80b

Date of Application: 3 April 1958

Publication of
application and issue
of Auslegeschrift: 8 September 1960

METHOD OF FABRICATING SILICON CARBIDE SHAPES

Applicant: Wacker-Chemie GmbH, Munich 22, Prinzregentstr. 20

Dr. Eduard Enk and Dr. Julius Nickl, Burghausen (Obb.), are named
as the inventors.

The problem of the technical preparation of silicon carbide for ordinary applications may be regarded as adequately solved. The resulting product, however, can not be employed directly for semiconductor purposes, especially if components with a definite shape are needed. Ordinarily, silicon carbide can only be produced in lumps, the shape of which is difficult to control.

It is known that silicon carbide or boron carbide shapes can be made by embedding objects formed from carbon in finely powdered silicon or boron carbide, a mixture of carbon and sand or boric acid, or pure silicon, and subjecting the bodies to a firing process.

In another method carbon is applied to molten silicon, or a mixture capable of yielding silicon carbide is applied to granulated silicon and heated.

A method has now been developed for making silicon carbide shapes by siliconizing preformed graphite or technical silicon



carbide bodies at a high temperature in a gas atmosphere containing silicon. The method is characterized in that the gas atmosphere containing silicon is produced by means of hydrogen-containing or hydrogen-free silicon halides, where necessary in the presence of hydrogen or compounds that split off hydrogen.

If one starts from technical silicon carbide, the generally formless lumps are shaped into platelets, rods or discs by breaking or grinding. Since graphite is easily molded into hollow shapes, if graphite is used as the starting material, the method makes possible the simple fabrication of intricate shapes such as tubes, vessels, etc., of highly pure silicon carbide.

The bodies made from technical silicon carbide are wholly or partially freed from silicon with chlorine or silicon tetrahalides at more than 1000°C. In this process all the undesirable impurities are simultaneously driven out of the crystal structure. If all the silicon is removed, there finally remains only highly pure graphite with the physical appearance of the given silicon carbide body. This wholly or partially silicon-free and extremely pure body is now resiliconized in a gas atmosphere until silicon carbide formation is complete. This can occur as follows:

Silicon is deposited on the graphitic body, with which it reacts, as a result of the thermal decomposition of hydrogen-containing silicon halides, e.g. silicochloroform, where necessary in the presence of hydrogen, or hydrogen-yielding compounds, such as methane.

A mixture of hydrogen-free silicon halides is brought to reaction with hydrogen or substances that split off hydrogen, e.g. hydrocarbons or silicochloroform, and the resulting silicon is directly deposited on the graphitic body with which it reacts to form silicon carbide.

A silicon subhalide, e.g. silicon dichloride, is produced from silicon and silicon tetrachloride and this dihalide is disproportionated on the graphitic body to silicon and silicon tetrahalide, the silicon reacting with the graphitic body to form silicon carbide.

In the latter case, it is necessary to work in a non-isothermal reaction vessel 2 resting in heat source 1, as shown in the drawing. On one side is the purified graphitic body 4 at a temperature T lower than that (T_1) of the highly pure silicon 3

from which it is spatially separated. The preferred agent for transferring the silicon 3 to the graphitic body 4 is silicon

tetrahalide. In the hot zone silicon and, for example, silicon tetrachloride vapor form gaseous silicon dichloride which reaches the colder graphitic body as a result of convection and diffusion and there decomposes into silicon and silicon tetrachloride. The reformed silicon tetrahalide then once more gives silicon dichloride with the hotter silicon 3.

The precipitated silicon, reacting in the nascent state, immediately forms silicon carbide with the graphitic body. If this process is carried out in a quartz tube, the siliconization can be accurately observed and interrupted at the moment that silicon carbide formation is complete. The same thing can be achieved with a dynamic arrangement, that is, by first allowing a stream of silicon tetrachloride, if necessary with hydrogen, to flow over highly pure and very hot silicon so as to form silicon dichloride, and then reacting the gaseous mixture of silicon dichloride and silicon tetrachloride at a somewhat lower temperature with the graphitic body.

CLAIMS

1. Method of fabricating silicon carbide shapes by siliconization of preformed graphite or technical silicon carbide bodies in a gas atmosphere containing silicon at a high temperature, characterized in that in order to produce the said silicon-containing gas atmosphere silicon halides containing hydrogen or free from hydrogen are used, where necessary in the presence of hydrogen or compounds splitting off hydrogen.

2. Method as defined under claim 1, characterized in that silicon subhalides are used for purposes of siliconization.

3. Method as defined under claim 1, characterized in that the said preformed bodies in technical silicon carbide are wholly or partially freed of silicon with chlorine or silicon tetrahalide at above 1000°C before siliconization.

REFERENCES

German Patents: Nos. 173 066, 183 133, 195 533;

US Patent: No. 2 691 605

[1 sheet of drawings annexed]

FARADAY TRANSLATIONS
15 PARK ROW
NEW YORK 38, N. Y.